Solvents for Extracting Nicotine from Aqueous Solutions

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The distribution coefficients of nicotine between water and several organic solvents at 10°, 25°, and 40° C. are reported. Chloroform is the most efficient solvent of those studied. Ethylene dichloride, chlorobenzene, and o-dichlorobenzene offer possibilities of improvement over kerosene as a solvent for recovery of nicotine in industrial processes.

SEVERAL organic solvents have been used to extract nicotine from aqueous solutions. Claffey *et al.* (*3*) studied the distribution coefficient of nicotine between water and Deobase at several temperatures and concentrations from an industrial process viewpoint. Norton (*6*, *7*) used petroleum and vegetable oils to determine their suitability for use in nicotine insecticidal sprays. Askew (*1*) used various petroleum distillates and found that those containing a higher proportion of aromatic compounds were more efficient. Hope *et al.* (*4*) used kerosene; Reilly *et al.* (*8*) used trichloroethylene; and Kolosovskiĭ *et al.* (*5*) studied several different organic solvents. Kerosene is used most commonly in industrial practice.

The distribution of nicotine between water and organic solvents is important in the industrial recovery of nicotine from tobacco wastes and in establishing the suitability of these solvents for insecticidal sprays. It is also important in predicting separation for analytical procedures such as the Craig countercurrent method. The work reported here was undertaken to evaluate the efficiency of reasonably available solvents and to compare them with kerosene and Deobase. Since temperature influences the distribution coefficient markedly, the measurements were made at 10°, 25°, and 40° C.

EXPERIMENTAL

The nicotine was obtained from a commercial grade (99% alkaloid). It was purified by two separate distillations in vacuo. The resulting nicotine was colorless, odorless, boiled at 133° to 135° C. at 32 mm. of pressure, and had a refractive index of $n_D^{25°} = 1.5240$. Titration against standard hydrochloric acid showed that it was substantially pure.

Because the distribution coefficient between water and kerosene is almost constant at concentrations from 0.01 to 5% of nicotine by weight in the aqueous phase (*3*, *5*) and since 2% is about the maximum that can be expected in commercial recovery of nicotine, an approximately 2% solution was used. Technical grades of organic solvents were used in most instances.

Fifty milliliters each of the aqueous nicotine solution and of the organic solvent were thoroughly mixed in an Erlenmeyer flask. The flask was stoppered and placed in a constant temperature bath maintained at the desired temperature, ±0.5° C. The flasks were shaken vigorously at intervals during the 48 hours they were maintained in the bath. At the end of this period, the layers were allowed to separate and duplicate samples of both layers were removed for analysis.

The nicotine was determined in the aqueous phase by titration against standard hydrochloric acid; methyl red was used as indicator. Water was added to the organic phase before titration. In several instances it was impossible to titrate the organic phase either because of color of the solvent or slight impurities. In these cases the nicotine in the organic phase was calculated by difference between the known amount originally placed in the flask and the amount found in the aqueous phase. Occasional checks against the titration method made by silicotungstic acid precipitation showed excellent agreement.

Table I gives the distribution coefficients, K. They were calculated from the equation $K = C/C'$, where C represents the concentration of nicotine in the aqueous phase and C', the concentration in the organic phase, both in grams per liter.

TABLE I. DISTRIBUTION COEFFICIENT OF NICOTINE BETWEEN WATER AND CERTAIN ORGANIC SOLVENTS

Solvent	Distribution Coefficient, C/C'[a], at 10° C.	25° C.	40° C.
Chloroform	0.016	0.013	0.008
Ethylene dichloride	0.104	0.068	0.057
Undecanol	0.154	0.082	0.062
Chlorobenzene	0.147	0.083	0.053
o-Dichlorobenzene	0.159	0.093	0.057
Benzene	0.187	0.104	0.075
Carbon tetrachloride	0.193	0.116	0.074
Nitrobenzene	0.223	0.124	0.117
Toluene	0.243	0.137	0.079
1,4-Dichlorobutane	0.208	0.142	0.642
p-Cymene	0.214	0.148	0.133
Butyl acetate	0.382	0.166	0.121
Xylene, A.C.S.	0.338	0.181	0.234
Dichloroethyl ether	0.251	0.181	0.689
sym-Tetrachloroethylene	...	0.181	0.107
Cyclohexane	1.160	0.557	0.268
n-Heptane	...	0.942	0.526
Kerosene[b]	...	1.11	...
Deobase[c]	2.84	1.25	0.63

[a] C is the concentration in the aqueous phase; C', concentration in the solvent phase.
[b] Values taken from Norton (*6*).
[c] Values taken from Claffey *et al.* (*3*).

DISCUSSION

All the solvents investigated are more efficient for extraction of nicotine from aqueous solutions than is kerosene or Deobase. Deobase is a refined grade of kerosene which has been treated to remove the unsaturates. The results show that it is slightly less efficient as an extraction solvent for nicotine than is kerosene. This agrees with the findings of Askew (*1*). Claffey *et al.* (*3*) give the distribution coefficient of nicotine between water and Deobase as 0.75, 0.42, 0.30, 0.33, and 0.23 at temperatures of 35°, 50°, 65°, 80°, and 98° C., respectively. Even at these elevated temperatures, Deobase does not compare favorably with most of the solvents studied here.

The criteria for a good solvent are K value, flammability, toxicity, solubility in water, and cost. From a practical standpoint, ethylene dichloride could be used for recovery of nicotine from dilute solutions. Smaller extraction columns or reduced amount of solvent could be used, as compared with those used at present. Chloroform, although the most efficient of the solvents investigated, has appreciable solubility in water and a high initial cost. Undecanol could not be used when the solvent is scrubbed with sulfuric acid to remove the nicotine because formation of a sulfate and its associated detergent action would result in emulsions. Chlorobenzene and *o*-dichlorobenzene offer possibilities,

for their cost is relatively low and their solubility in water is not appreciable.

In general, the efficiency of these solvents is greater at the higher temperatures. Three exceptions are 1,4-dichlorobutane, xylene, and dichloroethyl ether, which are more efficient at 25° than at 10° or 40°. This phenomenon also occurred in the case of toluene in Bowen's (*2*) study of the distribution of anabasine between water and certain organic solvents. With nicotine, however, the extraction efficiency of toluene increased with temperature. Bowen also found that chloroform and ethylene dichloride were better solvents than kerosene for extraction of anabasine from aqueous solutions.

LITERATURE CITED

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